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IS 8624 (1995): Method for determination of swelling index of iron ore pellets [MTD 13: Ores and Raw Materials]

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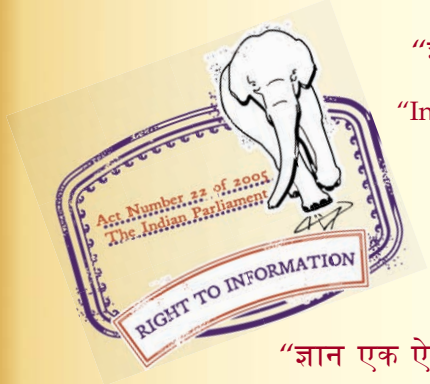

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(पहला पुनरीक्षण)

Indian Standard

METHOD FOR DETERMINATION OF SWELLING INDEX OF IRON ORE PELLETS

(First Revision)

UDC 622·341·1-188 : 620·192·53

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

June 1995

Price Group 5

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Ores and Raw Materials Sectional Committee had been approved by the Metallurgical Engineering Division Council.

It is well established fact that iron ore pellets swell during reduction. The excessive swelling of pellets may have an adverse effect on operation of a blast-furnace. Since swelling is an inherent property of pellets, a blast furnace operator should know the extent of swelling of pellets. This standard gives a method for determining the swelling index of indurated iron ore pellets.

This revised standard now covers two methods for determination of Swelling index of iron ore pellets. This has been done to harmonize the revised standard with that of ISO requirements. The test methods are technically equivalent to ISO/DIS 4698 which would facilitate evaluation of Indian iron pellets as per International Standards.

In the formulation of this standard, assistance has been derived from the following publications:

ISO/DIS 4698 'Iron ore pellets — Determination of relative free Swelling index', issued by the International Organization for Standardization (ISO).

JIS 8715 : 1965 'Method for measuring the Swelling index of iron ore pellets', issued by the Japanese Institute of Standardization (JIS).

In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHOD FOR DETERMINATION OF SWELLING INDEX OF IRON ORE PELLETS

(First Revision)

1 SCOPE

1.1 This standard covers the method for measurement of relative free swelling index of indurated iron ore pellets under isothermal reduction by two methods:

a) Mercury method, and

b) Oleate - Kerosene - Gakushin method (OKG Method)

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1992	Water for general laboratory use (*second revision*)
1405 : 1982	Methods of sampling of iron ore (*second revision*)

3 TERMINOLOGY

For the purpose of this standard, the following definitions shall apply.

3.1 Free-Swelling

A volume increase of iron ore or agglomerates which occurs during reduction under unconstrained conditions.

3.2 Free-Swelling Test

The determination of the unconstrained volume increase of iron ore pellets which occurs during reduction under specified conditons.

3.3 Free-Swelling Index

A relative measure of the volume increase of iron ore pellets which occurs during the free-swelling test. The index is expressed as a percentage.

4 TEST METHOD

4.1 Outline

After the volume of all pellets, of specified size range, selected for the test, is measured, they are placed into the sample holder which is then introduced in the reduction tube. The sample is reduced, isothermally, with specified reducing gas at a specified flow rate, at the specified temperature, for a specified period. The volume of all the pellets is measured, after reduction. Swelling index is calculated from the change of volume of pellets that occurs after reduction.

4.2 A representative test sample of about 2 kg shall be obtained from the physical test sample as per IS 1405 : 1982. The sample shall be screened to obtain pellets in the size range 10·0 to 12·5 mm. About 75 pellets shall be selected from these pellets for the test. The pellets selected shall be whole pellets. Each test sample shall consist of 18 pellets collected from about 75 pellets selected above, dried at 105 $\pm$ 5°C for at least 60 minutes.

4.3 Apparatus and Instruments (*see* Fig. 1, 2, 3)

a) Electric furnace, having heating capacity sufficient to maintain the entire test portion at 900 $\pm$ 10°C.

b) Reduction tube, made of non-scaling heat-resistant metal to withstand a temperature higher than 900°C. The internal diameter of the reduction tube shall be 75 $\pm$ 1 mm. The reducing gas shall be preheated before entering the test portion so as to maintain the test portion at 900 $\pm$ 10°C throughout the test period (*see* Fig. 2).

c) Test sample holder, made of non-scaling, heat resistant metal like INCONEL 600 to withstand a temperature higher than 900°C. It shall have an arrangement for holding 18 pellets, six pellets at each level, in three levels. The distance between the levels shall not be more than 30 mm and the pellets shall be so placed that the gap between adjacent pellets is not less than 10 mm.

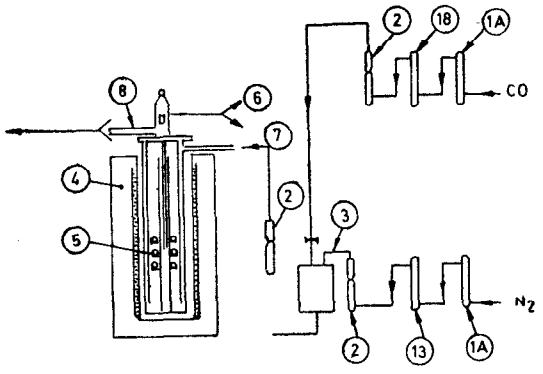
d) Suitable thermocouple and pyrometer for measuring temperature for ex: Pt/Rh or Chromel/Alumel.

e) *Volume Measuring Apparatus* — A typical apparatus for measuring the volume of the sample is shown in Fig. 3. With respect to the balance system, the part which is to be immersed in the mercury shall be given such a surface which will not alloy with the mercury. The float of

the balance system shall be made of a material which has a specific gravity slightly less than the apparent specific gravity of the ore pellets. The depth of the mercury vessel shall be about 150 mm. The minimum unit of the balance weights being used shall be 0·01 g.

f) *Weighing Device* — The minimum unit of the weighing device shall be less than 0·01 g.

g) *Thermometer* — The graduation of the thermometer shall be in units of 0·5°C or less.



Test device

1 Gas cylinders
2 Flow meters
3 Mixing vessel
4 Furnace
5 Test portion
6 Thermocouple
7 Gas inlet
8 Gas outlet

FIG. 1 EXAMPLE OF REDUCTION TEST APPARATUS

4.4 Gas to be Used

a) *Reducing Gas* — Dried and highly de-oxidised reducing gas shall contain (30 ± 0·5) % (*v/v*) CO and (70 ± 0·5) % (*v/v*) N_2. Impurities in reducing gas shall not exceed 0·2% (*v/v*) each of H_2, CO_2. Moisture and oxygen shall not exceed 0·1 % (*v/v*).

b) *Inert Gas* — Dried and highly de-oxidised N_2 or Argon shall be used.

4.5 Procedure

a) Determine the total volume of test portion, that is, all the 18 number of pellets (*see* Note).

b) Place six pellets each, on each of the three levels and place the sample holder in the reduction tube. Insert the reduction tube into the furnace.

c) Connect the reduction tube to the gas circuit.

d) Pass inert gas through the reduction tube at a flow rate of 10 l/min and commence the heating. When the temperature of the test portion approaches 900°C increase the flow rate of the inert gas to 15 l/min. Maintain the test portion under these conditions until the temperature is constant at (900 ± 10)°C.

e) Introduce the reducing gas to replace the inert gas at a flow rate of 15 l/min and continue reduction for 60 minutes at (900 ± 10)°C.

f) After reduction, replace reducing gas with inert gas at a flow rate of 5 l/min. Remove the reduction tube from the furnace and continue cooling while the inert gas is still flowing.

g) After cooling to room temperature, remove the sample from the reduction tube and determine its total volume (*see* Note).

NOTE — The volume of sample shall be measured as follows:

a) Weigh each piece separately.

b) Immerse the sample holder of the balance system into the mercury, place the weights on the weight pan until the needle tips touch the surface of the mercury. Push the balance rod gently by finger downwards. Then let it stand free, and confirm whether the needle tips touch the surface of mercury as in the initial trial or not. If the needle tips do not return to the initial position, repeat the same procedure until the needle tips touch the surface of mercury, by changing the weights and by turning the adjusting screw.

c) Gently lift up the balance system from the surface of mercury, insert the sample in the sample holder, repeat the procedure in (b) above, and record the amount of added weights.

d) Measure the temperature of the mercury in order to obtain its specific gravity at the testing temperature.

e) The volume of the ore or pellet, before and after reduction, shall be calculated to the second decimal place in accordance with the following formula:

$$V = \frac{W_1 + W_2}{S}$$

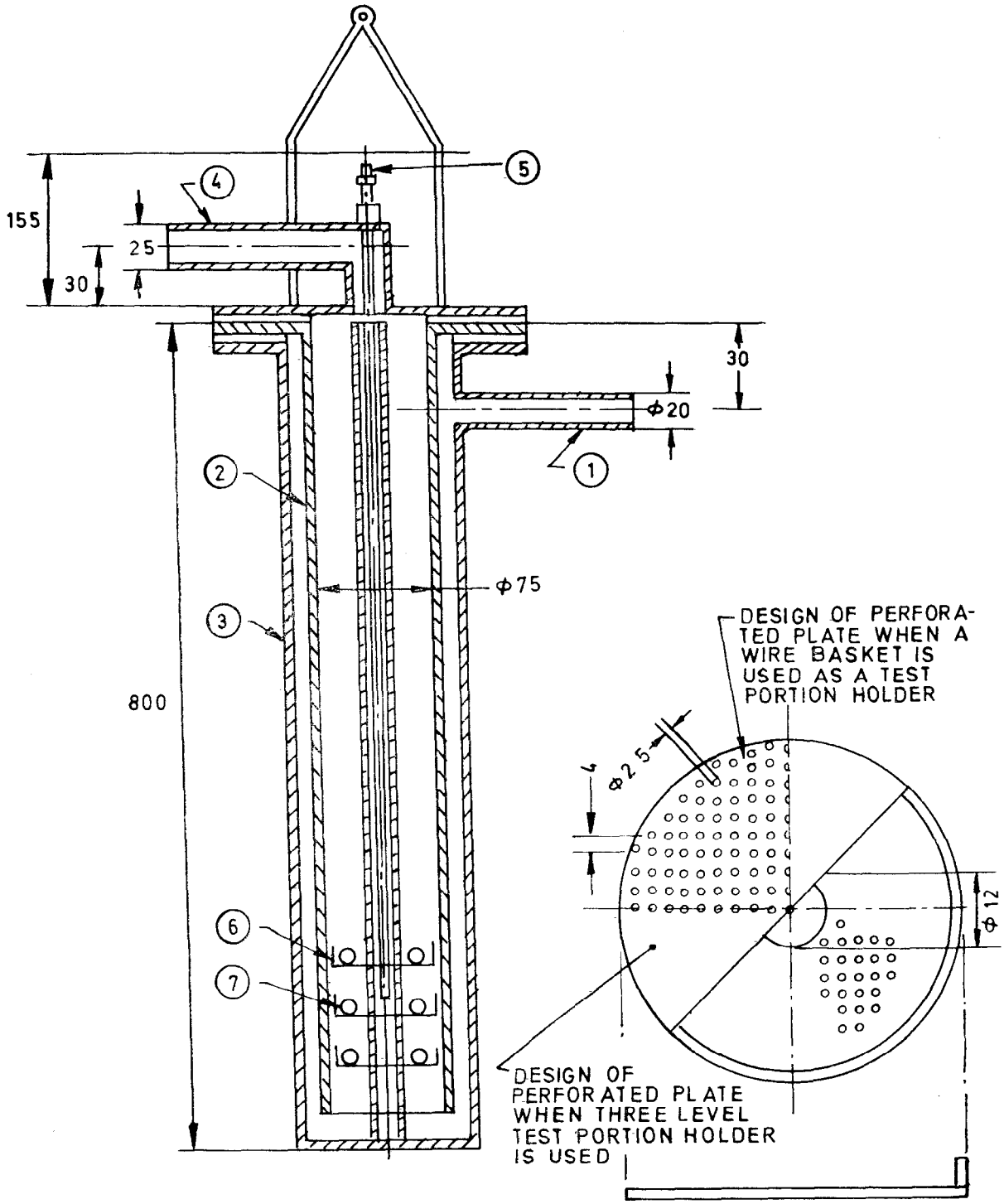
where

V = volume in ml of sample,
W_1 = mass in grams of the sample,
W_2 = mass in grams of the balance weights applied at the time of testing, and
S = specific gravity of mercury at the tested temperature.

4.6 Calculation of Swelling Index

The free swelling index, as a percentage, is calculated, using the following formula, rounded to first decimal place.

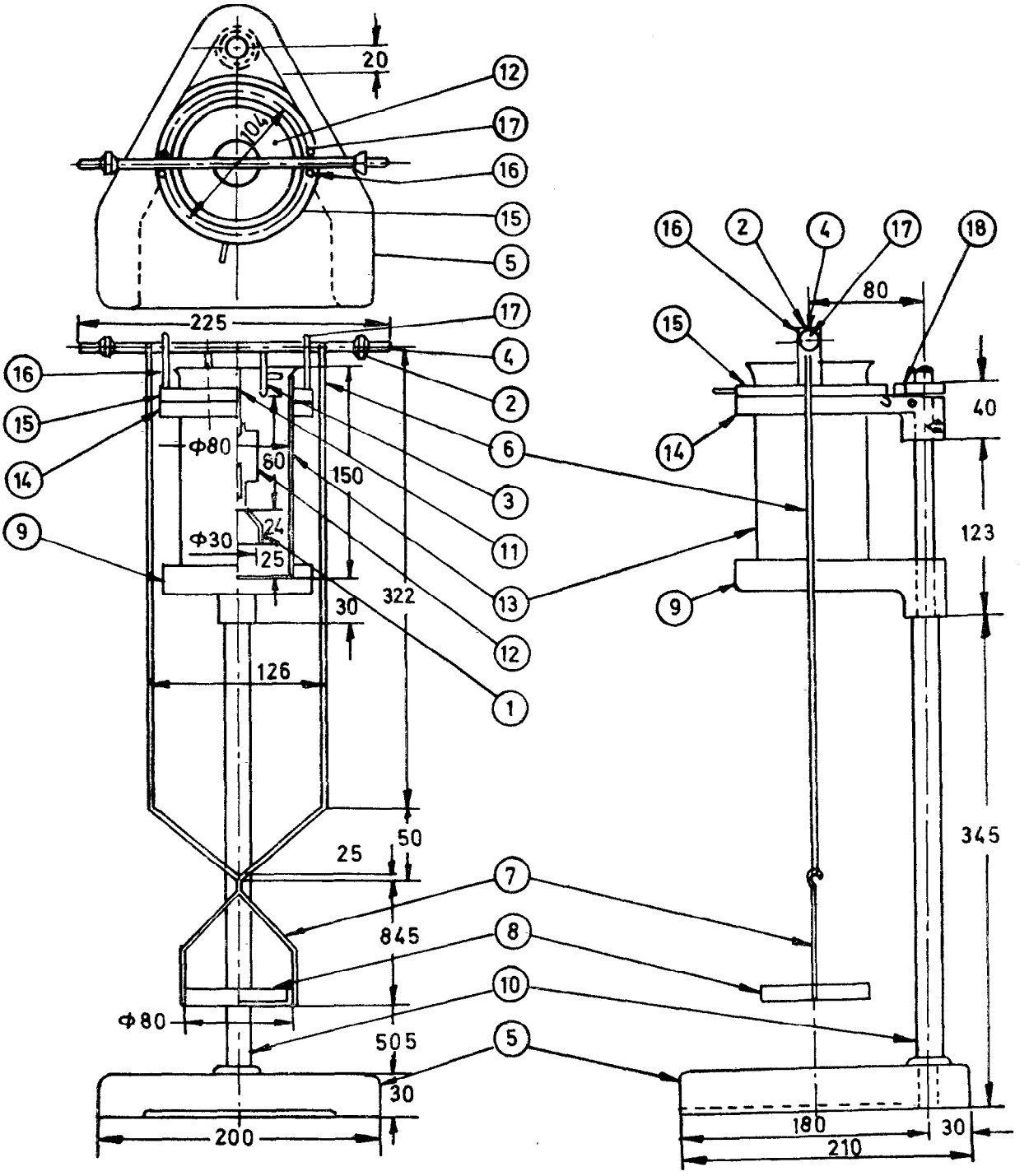
$$\text{Free-swelling index} = \frac{V_1 - V_0}{V_0} \times 100$$



Reduction tube

1 Gas inlet
2 Inner retort
3 Outer retort
4 Gas outlet
5 Thermocouple
6 Test portion holder
7 Test portion

FIG. 2 EXAMPLE OF REDUCTION TUBE WITH TEST PORTION HOLDER



No.	Name of Particle	No.	Name of Particle
1	Sample holder	2	Adjusting screws
3	Needles	4	Balance rod
5	Base	6	Suspending frame
7	Suspending frame for pan	8	Weights pan
9	Support for mercury vessel	10	Column
11	Suspending bar	12	Float
13	Mercury vessel	14	Instrument clamp
15	Fixing arm	16	Fixing pin
17	Supporting rod	18	Supporting plate

FIG. 3 AN EXAMPLE OF APPARATUS FOR MEASURING THE VOLUME OF PELLET

where

V_0 = volume in millilitres of the test portion before reduction, and

V_1 = volume in millilitres of the test portion after reduction.

4.7 Number of Tests

The test shall be carried out on duplicate test samples. If the difference between the paired results meets the permissible tolerance given in **4.8**, the test is terminated. If not, the flow sheet given in Annex A shall be followed.

4.8 Permissible Tolerance (*r*)

For a paired test, the difference between the two individual results shall be less than 30 percent absolute, for the free-swelling index.

4.9 Expression of Results

The final free-swelling index, expressed as percentage, as per Annex A, shall be expressed to one decimal place.

5 OKG METHOD (OLEATE-KEROSENE—GAKUSHIN METHOD)

5.1 Principle

After forming a hydropholic thin film on the surface of pellets by sodium oleate aqueous solution and stabilizing the film by kerosene, the weight of pellets both in air and water shall be obtained.

The volume of pellets shall be calculated on both weight of pellets and density of water.

5.2 Quality of Reagent

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070 : 1992) or ion exchange water shall be employed for the test. It shall be newly prepared whenever it is needed.

5.3 Apparatus

5.3.1 *Container*

A container of suitable size and material (like Inconel 600) in which the cage carrying pellets can move freely to hold sodium oleate aqueous solution.

5.3.2 *Cage*

5.3.2.1 *Cage for immersion in reagents*

The cage for immersion in reagents of suitable material (like Inconel 600) and size, where pellets can be stacked in 2 or 3 layers.

NOTE — The pellets be stacked in such a way as to avoid the breakage of pellets by a shock which is caused by the failing of the pellets to the bottom of the container.

5.3.2.2 *Cage for immersion in water*

It is to be used for immersing pellets in water and weighing the sample in this condition.

It shall be designed to minimize the contact surface area with the pellets so that the gas bubbles do not adhere to the cage.

NOTE — A typical example of glass made cage is shown in Fig. 4.

5.3.3 *Absorbent Container*

An absorbent container consists of one pair of sponges whose surface is covered with gauze.

It functions as agent absorbing froths of a reagent which remains on the surface of pellets immersed in reagents.

The gauze should be used in four folds (*see* Fig. 5).

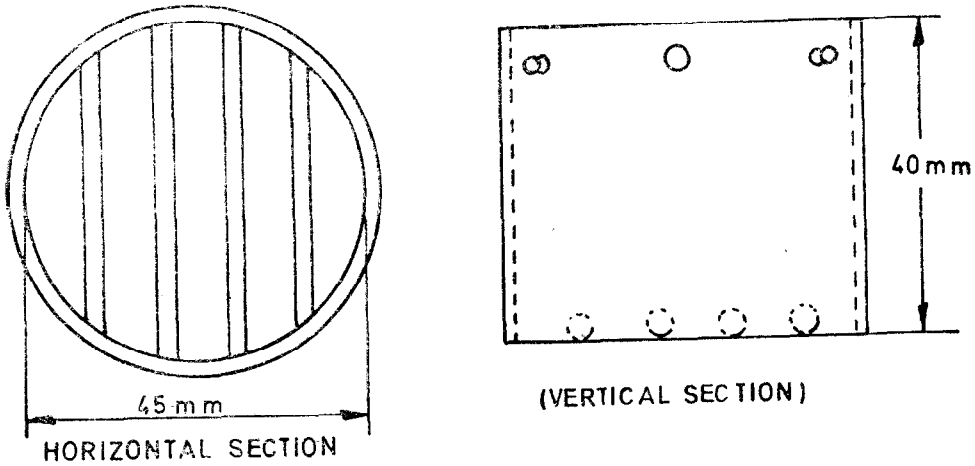


FIG. 4 AN EXAMPLE OF GLASS MADE CAGE FOR IMMERSION IN WATER

The sponges shall be selected for the sample to be laid stable and the surface area of it shall be selected to fit the sample numbers (*see* Fig. 5).

More than two pair of containers should be prepared for each reagent.

5.3.4 *Weighing Balance*

A weighing balance of 5 kg capacity and sensitivity 0·01 g.

5.3.5 *Suspensionary Line*

A thread (about 0·3 mm in diameter) with which a cage for immersion in water is suspended from the balance.

5.3.6 *Beaker*

It is used for weighing pellets in water and its capacity should be large enough not to contact the cage.

The base plane area of the beaker shall be large enough not to remarkably change the water level when weighing the sample weight.

5.3.7 *Stand*

A wooden or metal stand shall be used for keeping the beaker on it.

NOTE — A plastic stand (for example, acrylic resin made) should not be used so as not to be affected by static electricity.

5.3.8 *Thermometer*

A thermometer is graduated in 0·5°C or below.

5.4 Reagents

5.4.1 *Sodium Oleate Aqueous Solution*

(0·1 mol/l) — freshly prepared.

5.4.2 *Kerosene*

NOTE — Fresh kerosene of known specific gravity be used for each new determination.

5.5 Procedure

5.5.1 Place pellets in the cage (sodium oleate aqueous solution) and immerse, the cage with the pellets in the sodium oleate solution, for 30 min (*see* Fig. 6).

Shake the cage, to expedite the removal of the bubbles of air adhering to the sample surface.

5.5.2 Raise the cage and take out the pellets immersed in the solution and line up apart from each other in one pair of absorbent containers and then press the absorbent containers down slightly by hand for 10 s to absorb

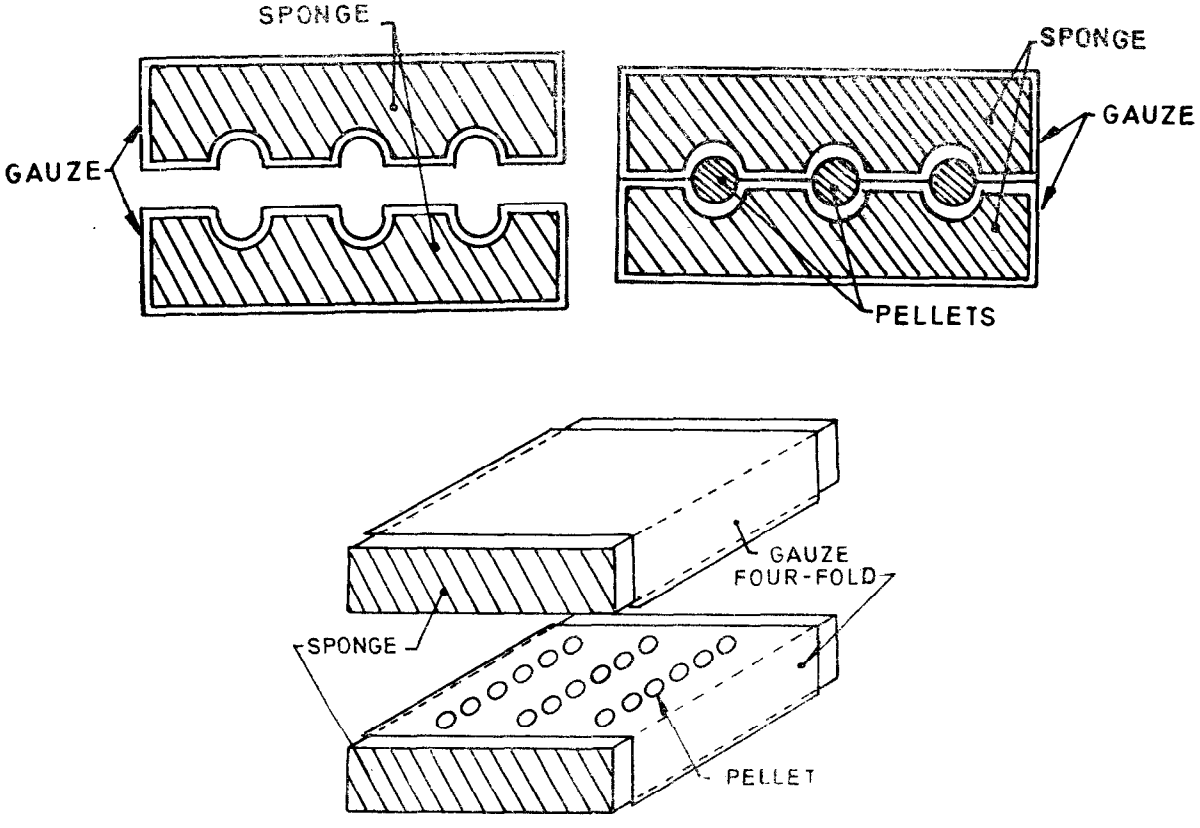


FIG. 5 ABSORBENT SPONGES

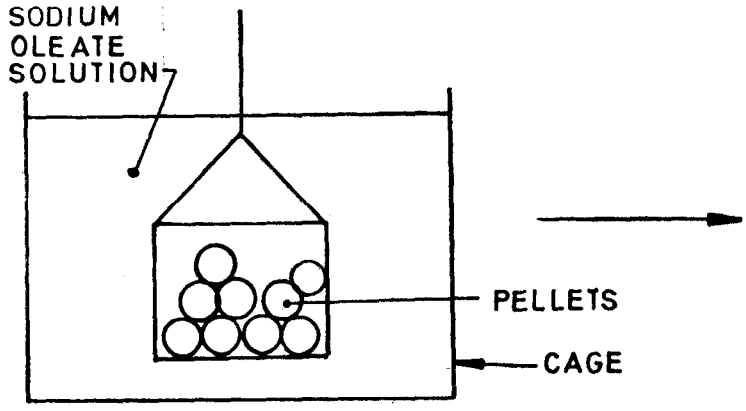


FIG. 6 GENERAL VIEW OF IMMERSION IN REAGENTS

the froths of sodium oleate adhering to the pellets.

Next, rotate the sample vertically to about 90° and line up another pair of absorbent containers and press the containers down again for 10 s to ensure that the bubbles are removed from the whole surface of the pellets.

5.5.3 Place the pellets as treated in the cage for kerosene and immerse the cage with the pellets in kerosene for about 10 s (*see* Fig. 7).

5.5.4 Raise the cage and take out the pellets immersed in kerosene and put it in the absorbent containers to absorb the froths of kerosene according to the procedures indicated in **5.5.3**.

5.5.5 Place the pellets as treated at **5.5.4** in the cage for immersion in water and suspened the cage with the pellets from the balance (*see* Fig. 8).

Weigh the sample accurately after 1 min while it is immersed in water (m_1).

5.5.6 Remove the pellets **5.5.5** from the cage and put them in the absorbent containers to absorb the water adhering to the sample surface.

5.5.7 Weigh the samples again (m_2).

5.5.8 After finishing the procedures at **5.5.6** suspend the cage from which pellets were taken out from the balance and immerse it in water. Obtain the weight in this stage of immersion of cage in water (m_3).

5.5.9 Measure the temperature of water in the beaker to know the density of water at that temperature.

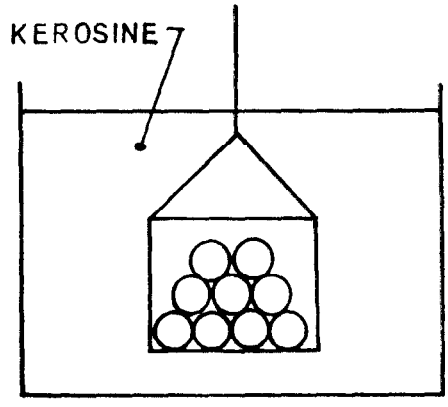


FIG. 7 GENERAL VIEW OF IMMERSION IN REAGENTS

5.6 Calculation of Volume

5.6.1 The volume of the pellets in millilitres is calculated to the third decimal place using the following formula:

$$V = \frac{m_2 - (m_1 - m_3)}{p}$$

where

V = volume, in millilitres (ml), of pellets;

m_1 = mass, in grams (g), in the state of immersing the cage with pellets in water;

m_2 = mass, in grams (g), of pellets;

m_3 = mass, in grams (g), in the state of immersing the cage in water; and

p = density, g/ml, of water with four significant figures.

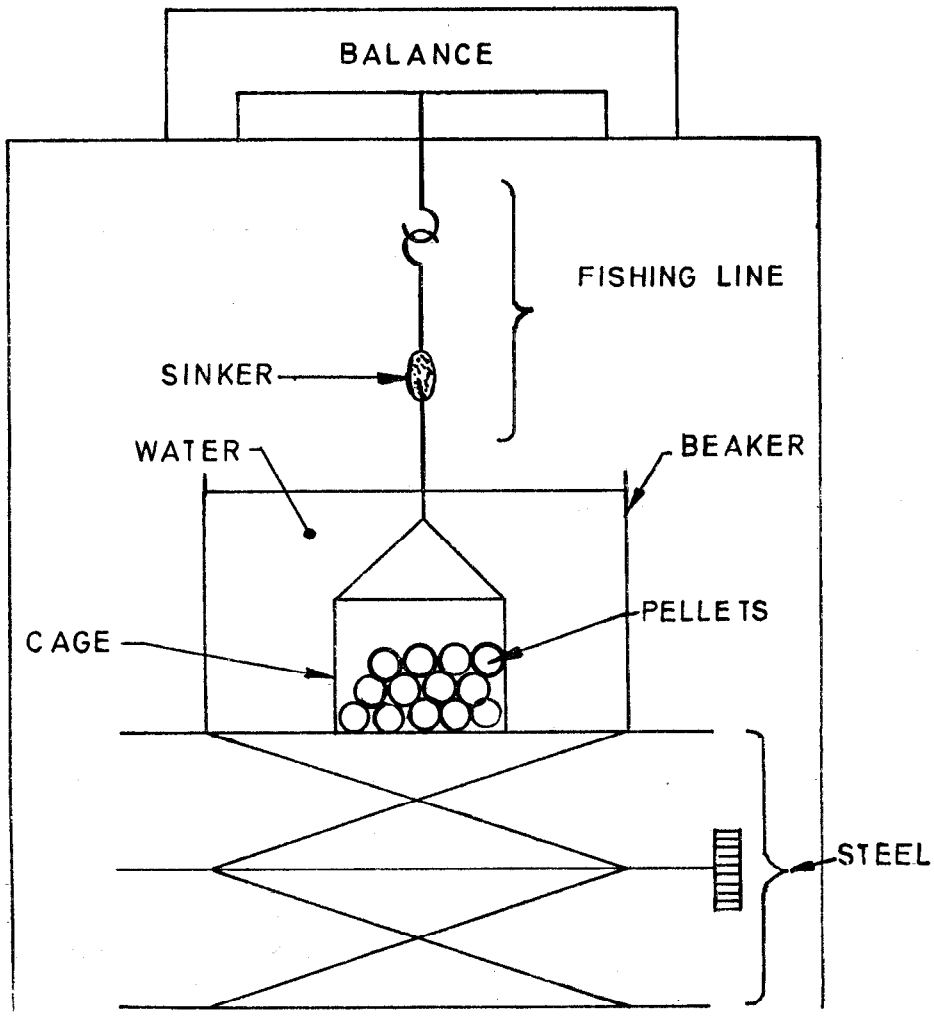


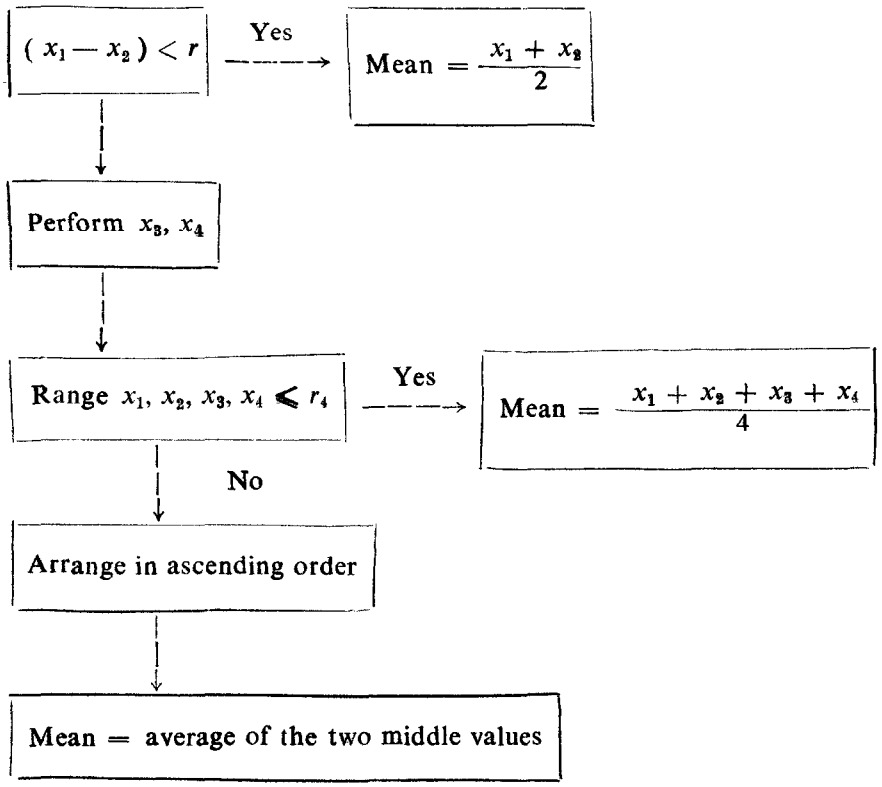
FIG. 8 GENERAL VIEW OF WEIGHT DETERMINATION IN WATER

ANNEX A

(*Clauses* 4.7 *and* 4.9)

FLOWSHEET FOR THE PROCEDURE FOR THE ACCEPTANCE OF THE TEST RESULTS

(Normative)



r = repeatability

$r_3 = 1{\cdot}20\ r$

$r_4 = 1{\cdot}31\ r$

Repeatability is the standard deviation within laboratories multiplied by 2·77.

x_1, x_2, x_3, x_4 are values of swelling indices of the test portions in that order.

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